				STUNFIGUT MAI ZUU.
ORM	PTO-13	90 (Modified) U.S. DEPARTMEN	IS OF COMMERCE PATENT AND TRADEMARK OFFICE	ATTORNEY'S DOCKET NUMBER
KBV I			TO THE UNITED STATES	206542US0PCT
			ED OFFICE (DO/EO/US)	U.S. APPLICATION NO. (IF KNOWN, SEE 37 CFR
			` ,	09/807997
, umm	722147	TONCERNING A FILIT	NG UNDER 35 U.S.C. 371 INTERNATIONAL FILING DATE	PRIORITY DATE CLAIMED
NIE		PCT/JP99/06159	5 November 1999	5 November 1998 (Earliest)
	E OF I	NVENTION		
			CIFIC TISSUE OF OILSEEDS OR C	EREALS AND FINELY CRUSHED
	VDE:			
		T(S) FOR DO/EO/US		
K 010	ini O.	ZAWA, et al.		
_		<del></del>	100 100 000 000 000	
Appi			ates Designated/Elected Office (DO/EO/US) the	
1.	$\boxtimes$		items concerning a filing under 35 U.S.C. 371	
2.			QUENT submission of items concerning a filir	
3.	Ø	This is an express request to be (9) and (24) indicated below.	gin national examination procedures (35 U.S.C	<ol> <li>371(f)). The submission must include itens (5), (6),</li> </ol>
4.	×		expiration of 19 months from the priority date	(Article 31).
5.	×	•	olication as filed (35 U.S.C. 371 (c) (2))	
			uired only if not communicated by the Interna	tional Bureau).
			ed by the International Bureau.	
		c.  is not required, as the	application was filed in the United States Reco	riving Office (RO/US).
6.	$\boxtimes$	An English language translation	of the International Application as filed (35 U	J.S.C. 371(c)(2)).
		<ul> <li>a.     is attached hereto.</li> </ul>		
		b.   has been previously se	ubmitted under 35 U.S.C. 154(d)(4).	
7.	$\boxtimes$	Amendments to the claims of the	e International Application under PCT Article	19 (35 U.S.C. 371 (c)(3))
e.		a.   are attached hereto (re	equired only if not communicated by the Intern	ational Bureau).
-		b.   have been communicated the communication in th	ted by the International Bureau.	
		c.  have not been made; l	owever, the time limit for making such amend	ments has NOT expired.
		d. Mave not been made a		
8.			of the amendments to the claims under PCT	Article 19 (35 U.S.C. 371(c)(3)).
9.	×		ventor(s) (35 U.S.C. 371 (c)(4)).	n n 1 nom
10.		An English language translation Article 36 (35 U.S.C. 371 (c)(5	n of the annexes of the International Preliminar )).	y Examination Report under PC1
11.		A copy of the International Pre-	iminary Examination Report (PCT/IPEA/409)	
12.	$\boxtimes$	A copy of the International Sea	rch Report (PCT/ISA/210).	
I	ems 1	13 to 20 below concern documen	nt(s) or information included:	
13.		An Information Disclosure Sta	tement under 37 CFR 1.97 and 1.98.	
14.		An assignment document for re	cording. A separate cover sheet in compliance	with 37 CFR 3.28 and 3.31 is included.
15.	×	A FIRST preliminary amendm	ent.	
16.		A SECOND or SUBSEQUEN	T preliminary amendment.	
17.		A substitute specification.		
18.		A change of power of attorney		
19.			te sequence listing in accordance with PCT Ru	
20.			international application under 35 U.S.C. 154	
21.			anguage translation of the international applica	tion under 35 U.S.C. 154(d)(4).
22.		Certificate of Mailing by Expre	SS Maii	
23.	A	Other items or information:	Notice of Bulleville.	
		PCT/IB/304 PCT/IB/308	Notice of Priority	
		Drawings ( 2 Sheets) Request for Consideration of	Documents Cited in International Search Re	port

J.S. Al	PLICA	TION N	O. (IF KNOWN, SEE	37 CFR	INTERNATIONAL AI			iO.	1		DOCKET NUMBER
		1	19/8 <b>0</b> 7	997	PCT/JP	99/0615	9			206542	US0PCT
24.	Т	he foll	owing fees are subn	nitted:.					CAI	CULATIONS	S PTO USE ONLY
BASIC	NAT	IONA	L FEE ( 37 CFR 1.	492 (a) (1) -	(5)):						
	Neithe international and In	r inter ational ternati	national preliminary search fee (37 CFR onal Search Report	examination 1.445(a)(2)) not prepared	fee (37 CFR 1.482) no paid to USPTO by the EPO or JPO	or 		\$1000.00			
$\boxtimes$	Interna USPT	ational O but l	preliminary examin international Search	ation fee (37 Report prepa	CFR 1.482) not paid to ared by the EPO or JPC	o )		\$860.00	1		
	but int	ernatio	onal search fee (37 C	CFR 1.445(a)	CFR 1.482) not paid to (2)) paid to USPTO		) 	\$710.00	l		
	but all	claim	s did not satisfy pro	visions of PC	CFR 1.482) paid to US T Article 33(1)-(4)			\$690.00			
	Internand al	ational l claim	s satisfied provision	s of PCT Art	CFR 1.482) paid to US icle 33(1)-(4)			\$100.00	_		
			ENTER AP	PROPRL	ATE BASIC FE	E AM	OU			\$860.00	
Surcha month:	rge of	\$130.0 the car	0 for furnishing the liest claimed priority	y date (37 Cl	FR 1.492 (e)).	□ 2	0	□ 30		\$0.00	
CL	AIMS		NUMBER	FILED	NUMBER EXT	RA		RATE	<u> </u>		
Total c	laims		20	- 20 =	0		х	\$18.00	<u> </u>	\$0.00 \$0.00	
	ndent o		3	- 3 =	0		х	\$80.00	├	\$0.00	
Multip	le Dep	endent	Claims (check if ap		ABOVE CALC	TIT AT	TO	NC -	1-	\$860.00	
	- Con	1 - in			R 1.27). The fees indic					\$00000	
i, re	educed	by 1/2	is small entity statu	s. (366 37 CI	K 1.27). The rees make					\$0.00	
27						SUB	TO		├	\$860.00	
Proces month	sing fe s from	e of \$1 the ear	30.00 for furnishing liest claimed priorit	the English y date (37 C	translation later than FR 1.492 (f)).	□ 2	0 _	□ 30 +		\$0.00	
A.					TOTAL NAT	IONA)	LF.	EE =		\$860.00	
Pee for	r record	ling th	e enclosed assignme	ent (37 CFR 1 neet (37 CFR	.21(h)). The assignme 3.28, 3.31) (check if a	nt must l pplicabl	be le).			\$0.00	
CON.					TOTAL FEES			ED =		\$860.00	
T.									Amo	ount to be: refunded	\$
over over										charged	\$
X a.	<b>X</b>	4 -1	eck in the amount of	6 5960	0.00 to cover the a	above fee	e ice	nclosed			
a. b.		Plea	se charge my Depos plicate copy of this	it Account N	o			of		to cover t	he above fees.
c.	$\boxtimes$	The	Commissioner is he	reby authoriz	ed to charge any additi	ional fees	whi	ch may be re	quired	, or credit any	overpayment
					A duplicate cor						
d.		Fees info	are to be charged to rmation should not	a credit care be included	d. WARNING: Inform on this form. Provide	ation on credit ca	this i	form may bee formation and	ome p d auth	orization on Pl	ro-2038.
NOTE 1.1376	E: Wh	ere an b)) mu	appropriate time l	imit under 3'	7 CFR 1.494 or 1.495 re the application to p	has not l	been tatus	met, a petiti	on to	revive (37 CFI	R
			ESPONDENCE TO						laru.	du Sacha	1
Telep	hone:		413-3000	_			S	GNATURE			
Fax:		(703)	413-2220		Surinder Sachar strauen No. 34,		N	orman F. C	Oblon		
				•			N	AME			
I			1000000		ı		2.	4,618			
1							_	EGISTRATI	ON N	UMBER	
			22	850				N	laz	4 2001	
		-					D	ATE			

#### IN THE UNITED STATES PATENT & TRADEMARK OFFICE

IN RE APPLICATION OF:

YOICHI OZAWA ET AL

: ATTN: NEW APPLICATION DIVISION

SERIAL NO: NEW US PCT APPN.

(Based on PCT/JP99/06159)

FILED: HEREWITH

FOR: METHOD FOR CLASSIFYING

SPECIFIC TISSUE OF OILSEEDS OR CEREALS AND FINELY

CRUSHED POWDERS

PRELIMINARY AMENDMENT

ASSISTANT COMMISSIONER FOR PATENTS

WASHINGTON, D.C. 20231

SIR:

Prior to examination on the merits, please amend the above-identified application as follows:

#### IN THE SPECIFICATION

Please amend the specification as follows:

Page 5, third line from the end to Page 6, line 2, replace the text in its entirety with the following:

--The term "specific tissue of oilseeds or cereals" means in this specification any tissues other than cotyledon, albumen, defatted soybean and wheat bran, which may be classified into their fine structure portions, including, for example, hulls (seed coat), germ, oil and fat-extraction cake, protein-extraction cake and the like.--

#### IN THE CLAIMS

Please amend the claims as shown in the marked-up copy and add new Claims 18-20 as follows:

- (Amended) A method according to Claim 1 wherein the classification is carried out based on the difference in specific volume or grain size of the crushed fine structure.
  - 9. (Amended) A method according to Claim 1 wherein the material is the fraction.
- 10. (Amended) A fraction derived from the fine structure portions of a specific tissue of oilseeds or cereals, which is obtained by the method according to Claim 1.
- 12. (Amended) An ultra-finely crushed powder according to Claim 11 wherein the material is selected from the group consisting of soybean hull, dry "okrara" or soybean pulp, soybean germ, corn germ cake, corn outer hull, rapeseed cake, and wheat germ.
- 14. (Amended) A method for producing ultra-finely crushed powder according to Claim 13 wherein the crushing is carried out in the dry state by using high-speed compressed air or high-speed compressed gas.
- (Amended) Food stuff comprising the ultra-finely crushed powder according to
   Claim 11.
- 16. (Amended) Culture medium for bacteria comprising the ultra-finely crushed powder according to Claim 11.
- (Amended) Filler for shaping containers comprising the ultra-finely crushed powder according to Claim 11.
- 18. (New) Food stuff comprising the ultra-finely crushed powder obtained by the method according to Claim 13.
- (New) Culture medium for bacteria comprising the ultra-finely crushed powder obtained by the method according to Claim 13.

 (New) Filler for shaping containers comprising the ultra-finely crushed powder obtained by the method according to Claim 13.

## REMARKS

Claims 1-20 are active in the present application. The claims are amended to remove multiple dependencies and to incorporate amendments entered during the international stage of this application. Support for Claims 18-20 is found in Claims 15-17. No new matter is added. An action on the merits and allowance of the claims is solicited.

Respectfully submitted,

OBLON, SPIVAK, McCLELLAND,

MAIER & NEUSTADT, P.C.

Norman F. Oblon Attorney of Record Registration No. 24,618

Daniel J. Pereira, Ph.D. Registration No. 45,518



(703) 413-3000 Fax #: (703) 413-2220

NFO:DJP\cct

I:\user\DJPER\206542US-pr.wpd

Marked-Up Copy
Serial No:
Amendment Filed on:

#### IN THE SPECIFICATION

Please amend the specification as follows:

Page 5, third line from the end to Page 6, line 2, replace the text in its entirety with the following:

--The term "specific tissue of oilseeds or cereals" means in this specification any tissues other than cotyledon, albumen, <u>defatted soybean</u> and wheat bran, which may be classified into their fine structure portions, including, for example, hulls (seed coat), germ, oil and fat-extraction cake, protein-extraction cake and the like,--

#### IN THE CLAIMS

Please amend the claims as follows:

- --8. (Amended) A method according to [one of Claims 1-7] <u>Claim 1</u> wherein the classification is carried out based on the difference in specific volume or grain size of the crushed fine structure.
- (Amended) A method according to [one of Claims 1-7] Claim 1 wherein the material is the fraction [obtained by the method according to one of Claims 1-7].
- 10. (Amended) A fraction derived from the fine structure portions of a specific tissue of oilseeds or cereals, which is obtained by the method according to [one of Claims 1-9] Claim 1.
  - 11. (Amended) An ultra-finely crushed powder having 50% grain size of 6 µm or

less, which is obtained from the material of a specific tissue of oilseeds or cereals.

- 12. (Amended) An ultra-finely crushed powder according to Claim 11 wherein the material is selected from the group consisting of soybean hull, dry "okrara" or soybean pulp, soybean germ, corn germ cake, corn outer hull, [whole soybean, defatted soybean,] rapeseed cake, and wheat germ.
- 14. (Amended) A method for producing ultra-finely crushed powder according to Claim [14] 13 wherein the crushing is carried out in the dry state by using high-speed compressed air or high-speed compressed gas.
- 15. (Amended) Food stuff comprising the ultra-finely crushed powder according to Claim 11 [or 12, or the ultra-finely crushed powder obtained by the method according to Claim 13 or 141.
- 16. (Amended) Culture medium for bacteria comprising the ultra-finely crushed powder according to Claim 11 [or 12, or the ultra-finely crushed powder obtained by the method according to Claim 13 or 14].
- 17. (Amended) Filler for shaping containers comprising the ultra-finely crushed powder according to Claim 11 [or 12, or the ultra-finely crushed powder obtained by the method according to Claim 13 or 14].

Claims 18-20 (New) .--

Method for Classifying Specific Tissue of Oilseeds or Cereals and Finely Crushed Powders

#### Technical Field

The present invention relates to a method for producing a fraction derived from the fine structure portions of a specific tissue such as hulls, germ, and cakes obtained by fat extraction or protein extraction of oilseeds (seeds rich in oil and fat) or cereals, comprising crushing material of the specific tissue and classifying the same.

The invention further relates to ultra-finely crushed powder having 50% grain size (the grain size that corresponds to 50% of an accumulated distribution curve of each grain size, which is the same as "50% diameter  $D_{50}$ ") of 6  $\mu$ m or less, to a method for producing ultra-finely crushed powder having 50% grain size of 10  $\mu$ m or less by crushing them in a dry state, and various composition comprising the ultra-finely crushed powder.

#### Background Art

A huge amount of oilseeds such as soybean, rapeseed, corn, and wheat is imported from abroad every year for the production of oil and fat, protein, starch and the like. Their cotyledon and albumen are mainly used for the above production. Oil component is usually extracted with n-hexane from cotyledon, albumen and germ. Soybean protein is further extracted from the resulting extraction cake with water.

The technique called a "dry millimg" is well known for separating in a dry state corn or wheat starch from their albumen, or soybean protein from cotyledon of the whole soybean or defatted soybean.

The Japanese Patent No. 2,803,853 discloses a method for the

separation of a protein-rich fraction and a fiber-rich fraction from wheat bran, in which the wheat bran was pulverized and classified into a fraction with a grain size of 300  $\pm$  25  $\mu m$  or less and 100  $\pm$  25  $\mu m$  or more, and a fraction with a grain size of 100  $\pm$  25  $\mu m$  or less.

The Japanese Patent Application laid open No.Hei.7-265000 discloses ultra-finely crushed powder that was obtained by preliminarily crushing a bran fraction of wheat, barley, rye, oat, adlay, sorghum and the like by means of a cutting and shearing mill, followed by the crushing with a jet mill-type crusher or a shearing mill. However, it is apparent that 50% grain size of the ultra-finely crushed powder was ten and a few microns or more as judged from a grain distribution chart described in the above reference.

On the other hand, materials derived from other tissues than the above bran, or the extraction cake of cotyledon, albumen and germ are commercially utilized as feed and fertilizers at a low price. Those materials are also used as a starting material for the extraction of polysaccharides (for example, the Japanese Patent Application laid open No. Sho. 64-62303 and the Japanese Patent Application laid open No.Hei.5-262802). And, they are finely milled to be used as food stuff (for example, the Japanese Patent Publication No. Hei. 3-69270 in which corn fiber was crushed into particles of 100 microns or less with a colloid mill-type mass colloider in a wet state; and the Japanese Patent Application laid open No.Hei.3-67595 in which raw "okara" or soybean pulp (a solid by-product in the production of soybean curd "tofu") was crushed into particle with an average diameter of 25 microns or less with a homogenizer in a wet state).

The Japanese Patent Publication No.Hei.3-69270 discloses an example wherein the corn fiber was crushed into 100 microns in length at its maximum measuring direction with the colloid mill-type mass colloider. As seen from the figures 1-5 in the same publication, it looks like that 50 % grain size of the crushed material was several tens microns or more.

Further, the Japanese Patent Publication No.Hei.3-67595 discloses an example wherein raw "okara" was crushed by means of the homogenizer into particles with an average grain size of 7.0 microns according to the measurement with a Coulter counter. However, the resulting crushed material is no more than an intermediate aqueous suspension that is obtained as a result of a pretreatment for the acceleration of decomposition of protein and fiber contained in water-insoluble dietary fiber in the processes of the production of water-soluble polysaccharides from water-insoluble dietary fiber. The crushed material was therefore subjected further to the decomposition treatment of protein and fiber contained therein. This means that the crushed material per se will not be used as food stuff and the like.

Thus, although some techniques are already known for finely crushing the specific tissues of oilseeds to be used as food stuff, all of them are carried out in the wet state.

On the other hand, the crushing using a Pinmill-type crusher in a dry state could only produce particles with an average gain size of several tens microns or more. Even the crushing using a ball mill-type crusher that was thought to be most effective in producing fine particles in the dry state could not produce particles with 50 % grain size of ten microns or less when material which is bulky and light or slippery on its surface such as the bean hulls was charged in the dry state. Thus, there is no example in which finely crushed particles with 50 % grain size of ten microns or less are actually obtained in the dry method, either.

According to an observation in a micro scale, the tissues of

oilseeds and cereals do not have a uniform structure, but a non-uniform structure such as that having layers (Watanabe, Tokio, Hashizume et al., "Soybean and Its Preparation" Kenko Co., p.6, 20 June 1987). Each of fine structure portions of the non-uniform structure is expected to have different chemical components and to show different physical properties.

Accordingly, if the tissues could be classified into fractions of the above portions, physicochemical properties that are not sufficiently apparent in the whole tissues might clearly appear.

However, except the above examples, there is no prior art that discloses the classification of the specific tissues of oilseeds and cereals into the fractions of fine structure portions.

The present inventors have completed this invention based on the finding that the specific tissue of oilseeds and cereals can be classified into fractions of the fine structure portions in accordance with the differences in their physicochemical properties such as specific volume and grain size pending on sensitivity to crushing, after being crushed into particles with several to several hundreds microns and classified by means of a sorting with air or a sieve.

By the way, it is said that our tongue can not feel roughness any more for particles with the grain size of about 20 microns or less. Accordingly, since the materials with an average grain size of several tens microns feel rough on our tongue and they are inferior in taste and suspending property, opportunities of their use as food stuff have been limited.

When suspension comprising the fine particles with the average grain size of several tens microns is used as a starting material for fermentation, brewing or enzyme reaction, there occur some problems to be resolved for an industrial application. That is, spore-forming bacteria can not be completely sterilized during a sterilization step and sterilization efficiency will become low, and precipitation will occur in a pipe line for transporting the suspension. Further, some precaution are always needed against corruption since the crushing in the prior arts was carried out in the wet state.

Besides, when the material with the average grain size several of tens microns is used as a filler material for shaping a container, the surface of the resulting container will become rough, and its quality will be inferior to a container comprising ultra-fine powder of calcium carbonate as the filler.

The present inventors have tested various methods in order to crush the material of the specific tissue of oilseeds and cereals into particles with 50 % gain size of 6 microns or less. As a result, we have succeeded in crushing the above materials into ultra-finely crushed powder having 50% grain size of 6  $\mu m$  or less, by using a method in which the materials collide to the wall of an apparatus or with each other by means of a high-speed compressed air or gas (Ultra-fine Jet Pulverizer (Kurimoto Ltd.), a jet miser, a micronizer, and Kriptron (Kawasaki Juko Co.), and the like).

#### Disclosure of the Invention

The present invention relates to a method for producing a fraction derived from the fine structure portions of a specific tissue of oilseeds or cereals, comprising crushing and classifying material of the specific tissue.

The term "specific tissue of oilseeds or cereals" means in this specification any tissues other than cotyledon, albumen and wheat bran, which may be classified into their fine structure

portions, including, for example, hulls (seed coat), germ, oil and fat-extraction cake, protein-extraction cake and the like.

Their preferred examples include the hulls of soybean, corn outer hull, rape seed, and sesame seed; husk; germs of soybean, corn, wheat, and rice; defatted rice bran; corn germ cake; dry "okara" or soybean pulp; cakes of rape seed and sesame seed.

The material of the specific tissue may be preliminarily dried before the crushing in order to control its water content, or may be used in an intact condition. The materials may be subjected to an appropriate treatment or processing.

Two or more kinds of the specific tissues may be combined at appropriate ratio and used as the material in the present invention.

Crushing may be carried out in this invention by any way and apparatus well known in the art both in a dry and wet state. In the wet state, the material has to be dried after crushing. Usually, the Pinmill-type crusher making use of shearing stress, mass colloider, impact stress-type crusher, and jet a jet miser, are preferably used to crush the material so that the fine structure portions of each specific tissue may be well peeled and separated with each other to give their mixture.

Some kinds of the crushing methods may be combined and used in the present invention. And, crushing conditions such as a degree of the crushing may be selected optionally by those skilled in the art, depending on the kinds of materials, use and application of the desired fraction, the kind of classifying method and the like. For example, the material may be crushed into powder having 50 % grain size of about a few  $\mu m$  - several hundreds  $\mu m$ , i.e., of 5  $\mu m$  - 500  $\mu m$  so that their fine structure portions may be separated well.

classifying may also be carried out in this invention by any way and apparatus well known in the art as long as it utilizes the differences in their physicochemical properties such as specific volume and grain size. For example, it may include the sorting with air and the sieve. The jet mill-type crusher may perform both crushing and classifying simultaneously.

Classifying conditions such as a degree of the classifying (the number of the resulting fractions) may be selected optionally by those skilled in the art, depending on the kinds of materials, use and application of the desired fraction, the kind of crushing method and the like.

Still, as shown by the following examples, it is preferred that the crushed material is classified into a fine fraction having 50 % grain size of a few  $\mu m$  - several tens  $\mu m$ , i.e., of 2  $\mu m$  - 50  $\mu m$  and a high specific volume of 2.5 ml/g - 3.1 ml/g, and a coarse fraction having 50 % grain size of several tens  $\mu m$  - several hundreds  $\mu m$ , i.e., of 50  $\mu m$  - 500  $\mu m$  and a low specific volume of 1.6 ml/g - 2.3 ml/g, since their physicochemical properties that are not sufficiently apparent in the original whole tissues will very likely appear clearly.

Mixture of one or more kinds of the fractions thus obtained may be used as the material in the present invention to be further crushed and classified.

The present invention is also related to the fraction derived from the fine structure portions of the specific tissue, which is obtained by the above method.

Especially, the fraction having 50 % grain size of a few  $\mu m$  - ten and several  $\mu m$ , i.e., of 2  $\mu m$  - 15  $\mu m$  is preferred since it has excellent properties.

The fraction according to the present invention is not

necessarily derived from one kind of the fine structure portions, but may contain more tow or more kinds of the fine structure portions depending on the crushing and classifying conditions.

The present invention is further related to ultra-finely crushed powder having 50 % grain size of 6  $\mu m$  or less, preferably 4  $\mu m$  or less, which is obtained from the material of the specific tissue of the oilseeds or cereals.

The method for crushing of the materials may be optionally selected without any limitation by those skilled in the art as long as the desired 50 % grain size is obtained. As described before, the methods are preferred in which the material collides to the wall of an apparatus or with each other by means of a high-speed compressed air or gas, or by means of rotation at a high speed, using the jet mill-type crusher (Kurimoto Ltd.), the jet miser, the micronizer, and Kriptron (Kawasaki Juko Co.), and the like.

A classifying rotor may be combined with the above apparatus in appropriate conditions to obtain the ultra-finely crushed powder having a desired grain size more effectively.

The invention is therefore related to a method for producing ultra-finely crushed powder having 50 % grain size of 10  $\mu$ m or less, preferably 6  $\mu$ m or less, more preferably 4  $\mu$ m or less, comprising crushing the material of the specific tissue of oilseeds or cereals in a dry state.

Preferably, the crushing is carried out in the dry state by using the high-speed compressed air or gas.

The crushing conditions may be selected optionally by those skilled in the art, depending on the kinds of materials, the desired average grain size, and use and application of the resulting ultra-finely crushed powder.

The materials may be directly crushed, or may be preliminarily crushed with a mechanical crusher such as the Pinmill-type crusher to a grain size of several tens  $\mu m$  - several hundreds  $\mu m$ , for example, 50  $\mu m$  - 300  $\mu m$  so as to shorten the time for crushing into the ultra-finely crushed powder of the present invention.

Mixture of two or more kinds of the materials of the specific tissues may be subjected to the crushing method of the present invention. Alternatively, mixture of two or more kinds of the ultra-finely crushed powder that are derived from different materials may be included in the present invention. Mixing ratios in these mixtures may be optionally selected by those skilled in the art depending on their use and the like.

The ultra-finely crushed powder of the present invention is much more advantageous than the conventional finely-crushed powder having 50 % grain size of several tens  $\mu m$  - ten and several  $\mu m$  in the following aspects:

- properties as food stuff such as smoothness, water holding capacity, suspension-state holding capacity;
- 2)sterilization efficiency of the suspension and transportation efficiency in the pipe line when used as material for fermentation;
- extraction efficiency of various components contained in the ultra-finely crushed powder;
- reaction efficiency with enzymes, digestion and absorption efficiency, and feed efficiency when used as feed component;
- 5) quality of containers such as a food tray having a good biodegradablity, which comprises the ultra-finely crushed powder of the present invention as filler.

Accordingly, the present invention is also related to various kinds of compositions that comprise the ultra-finely crushed powder of the present invention, such as food stuff for various kinds of food (confectionery, bread, desserts such as ice cream, source, retort pouched food, oil and fat food, emulsified food, liquid food, dietary fiber-enriched food, low-calorie food, low-fat food, nutritional component-enriched food, animal food product, marine product and the like), a culture medium for bacteria in fermentation and brewing, filler for shaping containers including the food trays, and the containers comprising the filler.

# Brief Description of Drawings

Fig.1 shows charts of the grain size distribution of the ultra-finely crushed soybean hull obtained by the treatment with the Pinmill-type crusher two times (50 % grain size:75 µm)(A), the soybean hull-derived "fine" powder obtained by the sorting with air of the ultra-finely crushed soybean hull (A) (50 % grain size:12 µm)(B), and the soybean hull-derived "coarse" powder obtained by the same way (50 % grain size:163 µm)(C). In each chart, a bar graph represents frequency (%) scaled on the left vertical axis, and a line graph represents accumulation (%) scaled on the right vertical axis. A horizontal axis means grain size (µm).

Fig. 2 shows charts of the grain size distribution of various kinds of the ultra-finely crushed powder prepared in Example 7.

(a):soybean hull (50 % grain size:6.0 µm); (b):soybean hull (50 % grain size:3.4 µm); (c):dreid "Okara" (50 % grain size:3.1 µm); (d):corn germ cake (50 % grain size:5.0 µm); and (e):roasted soybean germ (50 % grain size:3.4 µm). In each chart, a bar graph represents frequency (%) scaled on the left vertical axis, and a line graph represents accumulation (%) scaled on the right vertical axis. A horizontal axis means grain size (µm).

Best Mode for Carrying Out the Invention

The value of "50% grain size" in the following examples was measured in ethanol by means of MICROTRAC II DRY SYSTEM (NIKKISO). The term "50% grain size" means the grain size that corresponds to 50% of an accumulated distribution curve of each grain size, which is the same as "50% diameter  $D_{40}$ ".

The T-N (Total Nitrogen) value was measured in Kjeldahl method with KUELTEC AUTO 1838 Analyzer.

The specific volume was determined as follows: the ultra-finely crushed powder was gradually added to a measuring cylinder (500 ml) while being vibrated until a level of 500 ml would not change any more, and the weight of the ultra-finely crushed powder comprised at the level of 500 ml was measured. The specific volume was calculated from the weight thus measured.

The present invention will be specifically described by referring to the following examples, which should not be construed to limit the technical scope of the present invention.

#### Example 1

Soybean hull was crushed with a Pinmill type crusher. In the case of Pallmann-Universal Mill PX, the crushing was done at a rotation rate of 17,000/ min. and a charged amount being about 60 kg/ hr. 50% grain size of the resulting crushed powder was 150  $\mu m$ . The second, third and forth (final) crushing in the same conditions gave the ultra-finely crushed powder derived from the soybean hull having 50% grain size of 75  $\mu m$ , 43  $\mu m$ , and 28  $\mu m$ , respectively (Table 1). They were then classified with the air sorting. The classifying conditions are summarized in Table 2 below. The results of the classification are shown in Table 3.

[Table 1]

	Times of Crushing	50% Grain Size
	one	150 μm
oybean	two	75
hull	three	(43)
	four	28

# [Table 2]

Material	50% Grain Size	Air Rate	Rotation in Sorting	Production Rate
Pinmill Crsushing (once)	150 μm	1 m³/min	3,000 rpm	1440 g/h
Pinmill Crsushing (twice)	75	the same as above		1280
Pinmill Crsushing (four times)	28	the same as above	5,000	975

[Table 3]

	Fractions by Classificat					on with the Air Sorting				
Materia	1	Fine (light) Powder				Coarse (heavy) Powder			der	
(Soybean h	u11)									
	50%	Color	50 %	Yield %	T-N%	Color	50 %	Yield	T-N%	
	Grain		Grain				Grain	%		
	Size		Size		l		Size			
Pinmill	150	White-	26 µm	37	5.65	Sepia	223 µm	63	1.64	
Crsushing (once)	μm	gray								
Pinmill	75	White-	12	36	6.16	Sepia	163	64	1.78	
Crsushing (twice)		gray				-				
Pinmill	28	White-	11	44	4.11	Sepia	77	56	1.31	
Crsushing (four tiems)		gray								
(		8	1	ı	1	11	E	1	1	

As shown in Table 3, each of the resulting powder was classified into a fine (light) powder fraction and coarse (heavy) powder fraction, each fraction having different values of 50% grain size and T-N. The fact that these two fractions have a different microscopic shape with each other seems to demonstrate the difference in their fine structure. The color of the crushed powder before the air sorting was pale sepia.

Analytical values of the contents of the fine and coarse fractions classified from the ultra-finely crushed powder obtained through the above four crushing procedures with the Pinmill-type crusher are summarized in Table 4. The specific volume of the fine powder was 2.73 ml/g, and that of the coarse powder 2.02 ml/g.

[Table 4]

Sample	Water %	Protein %	Fat & Oil	Fiber %	Ash %	Sugars %	Dietary Fiber %	Energy
Fine Powder	7.2	24.0	6.2	13.0	6.8	42.8	47.4	375 kcal
Coarse Powder	6.8	8.0	1.7	37.6	4.3	41.6	76.5	364

(Analyzed by Japan Food Analysis Center)

As shown in Table 4, the analytical values of the contents of water, protein, fat and oil, fiber, ash, sugars, and dietary fiber, and of energy were significantly different with each other, which means that the fine structure of the soybean hull was classified according to the present method.

Fig.1 shows the grain size distribution of the soybean hull-derived fine powder (specific volume: 2.84 ml/g) and the soybean hull-derived coarse powder (specific volume: 1.92 ml/g), which were classified with the air sorting from the ultra-finely crushed soybean hull obtained by the treatment with the

Pinmill-type crusher twice. Fig.1 demonstrates that the soybean hull can be classified, and a fraction obtained by the classification can be more finely classified again.

## Example 2

Dry "okara", corn germ cake and soybean germ were used as a starting material of the present invention.

Dry "okara" (freeze-dried raw "okara"), corn germ cake and soybean germ were pre-heated at 80 °C or more and crushed with the Pinmill-type crusher. The resulting products were then crushed with a jet mill-type crusher (air pressure:7kg/cm2, nozzle:3\phi, 11,500 rpm) while being classified with air sorting into a fine powder fraction that was discharged from the upper part of the crusher and a coarse powder fraction that remained in it. The results are shown in Table 5.

[Table 5]

	Material		Fraction					
	Pinmill Crsus	shing (once)	Fine Po	owder	Coarse Powder			
	50% T-N %		50%	T-N %	50%	T-N %		
	Grain Size		Grain Size		Grain Size			
Dry "Okara"	93 μm	6.06	3.1 μm	4.57	10.4	6.34		
Corn Germ Cake	361	2.72	5.0	3.18	24.0	2.56		
Soybean Germ	54	6.03	3.4	5.89	22.0	6.21		

The N-T value of the resulting fine powder was different from that of the starting material, demonstrating that the crushed material was classified into the fine powder and coarse powder that reflected a micro partial structure of the material.

## Example 3

The ultra-finely crushed soybean hull powder (50 % grain size: 75  $\mu$ m) obtained by the treatment with the Pinmill-type crusher twice under the same conditions as in Example 1 was classified with various kinds of a vibrating sieve having 60 - 400 meshes for 5 min. The weight of each resulting fraction was determined and their 50 % grain size was calculated. The results are shown in Table 6.

Table 6 shows that peaks in the weight are found in the fractions obtained with 250-400 mesh (50 % grain size: 39 µm) and 80-120 mesh (50 % grain size: 172 µm). If fine powder and coarse powder are divided into two fractions on the borderline of 400 mesh, the ratio between the two fractions will be 31:69, showing that it is possible to carry out the classification with the sieve according to the present invention.

[Table 6]

Meshes	Below 400	250-400	200-250	120-200	80-120	60-80	0ver 60	
Weiht %	3.6	27.2	12.5	18.8	19.1	12.2	6.6	100 %
50% Grain	18	39	64	125	172	247	386	
Size								

#### Example 4

The ultra-finely crushed soybean hull powder (50 % grain size:  $75 \mu m$ ) obtained by the treatment with the Pinmill-type crusher twice under the same conditions as in Example 1 was classified with the air sorting according the same conditions as in Example 1 to give a fraction of fine powder (50 % grain size: 12  $\mu m$ ) and a fraction coarse powder (50 % grain size: 163  $\mu m$ ).

Water-soluble protein and polysaccharides were then extracted

from the above fractions.

The powder (50 g) was mixed with water (500 ml), adjusted to pH 9, and stirred at a room temperature for 60 min for extraction, followed by centrifugation to remove contaminants. Supernatant was lyophilized to give a solid material. Table 7 shows the extract conditions, extraction rate and T-N values of the extracts.

As shown in Table 7, the fine powder has a higher extraction efficiency (yield) and T-N value than those of the whole part of the crushed soybean hull, demonstrating that the fine powder is more advantageous as a starting material for the extraction of the protein.

[Table 7]

Material	Yield of Extract in Water at pH 9, room temp., 60 min	T-N %
Crushed Soybean Hull Powder	1 2%	4.1
Fine powder	2 5	6.2
Coarse Powder	8	2.3

# Example 5

The ultra-finely crushed soybean hull powder (50 % grain size: 75  $\mu m)$ , the fraction of fine powder (50 % grain size: 12  $\mu m)$  and the fraction of coarse powder (50 % grain size: 163  $\mu m)$ , which were obtained in Example 4, were used as starting materials for the extraction of oil contained therein with n-hexane. The extracted oil was subjected to a thin layer chromatography and gas chromatography to determine a total amount of phytosterol according to a standard oil analysis test (Japan Oil Chemists Society).

The results summarized in Table 8 show that the oil content in the coarse powder was 1.7 %, whereas the total amount of phytosterol was 22.7 % that is about 56 times as high as that of a usual soybean oil. On the other hand, the oil content in the fine powder was 6.2 % that is about 3.6 times as high as that in the coarse powder, but the total amount of phytosterol was about one fifth of that in the coarse powder.

Thus, it is possible to classify the soybean hull crushed powder and to extract a specific and advantageous component locally existing in the hull part such as phytosterol from the classified products in a more effective way at a high concentration. The phytosterol is known to have a function to inhibit the absorption of cholesterol or to promote its discharge from a body. The coarse powder, which correspopnds to the fraction according to the present invention, is advantageous as a starting material for a oil component that is rich in phytosterol. The measurement of the total amount of phytosterol was carried out by JAPAN OILSTUFF INSPECTORS.

[Table 8]

Material	Oil Content %	Total Amount of Phytosterol %
Crushed Soybean Hull Powder	3.1	7.0
Fine powder	6.2	5.7
Coarse Powder	1.7	8.3
Normal Soybean	18 - 20	0.4

# Example 6

Each 20 g of the fraction of fine powder (50 % grain size: 12  $\mu$ m) and the fraction of coarse powder (50 % grain size: 163  $\mu$ m), which were obtained in Example 4, was mixed with water (200 ml) and subjected to the extraction with stirring at pH 4.5 and a room temperature for 60 min, followed by centrifugation to

remove solid components. The resulting extract was lyophilized and subjected to an amino acid analysis. The results in Table 9 show that the fraction of the coarse powder of the soybean hull according to the present invention is advantageous as a starting material for the extraction of protein that is rich in HO-Pro contained in the soybean hull since it has a significantly high content of HO-Pro in water extract.

[Table 9]

	HO-Pro (mole %)
Fine Powder	0.0
Coarse Powder	4.1
Soybean Globulin	0.0

## Example 7

The soybean hull was roughly cut into cubes of about 1-3 mm³ and preliminarily crushed with the Pinmill-type crusher. Dry "okara" (lyophilized flakes of raw "okara") and corn germ cake were preliminarily crushed with the Pinmill-type crusher. Roasted soybean germ was prepared by preliminarily heating the soybean germ cake at 80°C for 40 min, and preliminarily crushed with the Pinmill-type crusher. These materials were then crushed with Ultra-fine Jet Pulverizer(Kurimoto Ltd.). The crushing conditions and 50 % gain size of the resulting ultra-finely crushed powder are summarized in Table 10.

[Table 10]

Materi	al	Crushing Conditions	Results	
	50 % Grain Size(μm)	Rotation Rate and Air Pressure	50 % Grain Size(μm)	
Soybean Hull	1 - 3 x 10 <sup>3</sup>	10,000 rpm, 7 kg/cm <sup>2</sup>	6.0	
Soybean Hull	150	the same as the above	3.4	
Dry "Okara"	93	11,500 rpm, 7 kg/cm <sup>2</sup>	3.1	
Corn Germ Cake	361	6,000 rpm, 8 kg/cm <sup>2</sup>	5.0	
Roasted Soybean Germ	54	10,000 rpm, 7 kg/cm²	3.4	

As shown in Table 10, the values of about 3-6  $\mu$ m were obtained with respect to 50 % grain size for all of the materials used. Refer to the grain size distribution in Fig. 2 as well.

(1) Flaked and defatted soybean germ, (2)flaked and defatted soybean cotyledon and (3) dry and crushed "okara" were ultra-finely crushed using Kriptron KTE-type crusher (Kawasaki Heavy Industry Co.). The crushing conditions and the results are summarized in Table 11.

[Table 11]

No.	Material	Sizes	Rotation Rate	Air Rate (Nm³/min)	Feed Rate (Kg/hr)	Results (50 % Grain Size)
1	Flaked and Defatted Soybean Germ	0.1 - 5 mm long, 0.05 - 1.5 mm thick	10,400 rpm	8	15	6,4
2	Flaked and Defatted Soybean Cotyledon	0.1 - 7 mm long, 0.05 - 1.5 mm thick	10,400 rpm	8	15	6.7
3	Dry and Crushed "Okara"	10 μm (50 % Grain Size)	10,400 rpm	8	15	7.4

As shown in Table 11, the values of about 6-8 µm were obtained with respect to 50 % grain size for all of the materials used.

# Example 8

The suspendibility of the ultra-finely crushed powder obtained in Example 7 was evaluaed. After each ultra-finely crushed powder (1 g) was suspended in water (100 ml), the resulting suspension was moved to a measuring cylinder (100 ml) and allowed to stand at a room temperature for 24 hr. The level of the boundary between a transparent layer and an opaque suspension layer was recorded. For comparison, suspendibility of each of the powder crushed with the Pinmill-type crusher was measured in the same way.

As shown in Fig. 12, the boundary level of the crushed powder having 50 % grain size of several hundreds  $\mu m$  ~ several tens  $\mu m$  become 10 ml or less after 24 hr. On the other hand, the ultra-finely crushed powder having 50 % grain size of about 3  $\mu m$  ~ 6  $\mu m$  according to the present invention kept its boundary level around 95 ml even after 24 hr, demonstrating that its precipitating rate has been significantly decreased while its suspendibility has been significantly improved.

For comparison, the corn fiber crushed into particles of 100 microns or less with the colloid mill-type mass colloider in the wet state described the Japanese Patent Publication No.Hei.3-69270 was also subjected to the same experiment to show the boundary level of 18 ml.

## [Table 12]

Ultra-finely	Crushed Powder	Level of the	Crushed Powder	Level of the
Crushed Powder	With Jetmill	Boundary after	With Pinmill	Boundary after
	50% Grain	24 hrs (m 1)	50% Grain	24 hrs (m 1)
	Size (μm)		Size (µm)	
Soybean Hull	6.0	9 3	9 5	7
Dry "Okara"	3. 1	ું છે છે	93	8
Corn Germ Cake	5.0	9 7	3 6 1	6
Roasted Soybean	3.4	9 7	5 4	5
Germ				

## Example 9

The ultra-finely crushed powder obtained in Example 7 was used as food stuff.

(1)Hamburger: The difference in taste was compared and evaluated between a control hamburger and a test one comprising the ultra-finely crushed powder of soybean hull having 50 % grain size of 6.0 µm.

Mixed ground meat of pork and beef 40.1 (parts); soybean protein 2.8(parts); water 11.5(parts); soybean oil 2.8(parts); onion 17.2(parts); bread crumb 8.6(parts); milk 8.6(parts); egg 7.2(parts); salt 1.0(parts); pepper 0.06(parts); and nutmeg 0.05 (parts)were kneaded, shaped and heated to give a control product. A test product was prepared following the same recipe except that the soybean protein 0.5 was replaced with the same amount of the above ultra-finely crushed powder of soybean hull.

According to a sensory test by a panel consisting of eight persons, no panelist felt roughness for the test product, and the test product was evaluated to be superior to the control one in both hardness and no paste-like feeling. Furthermore, they evaluated that the test product was superior to the control one in binding property and shape-holding capability.

(Evaluation: 1(bad) - 5(excellent))

[Table 13]

	Results of Sensory Test						
	Hardness	No Paste-like Feeling	Total Feeling				
Control Product	2.8	1.5	2.7				
Test Product	3.1	1.7	3.1				

(2) Beverage: A test product was prepared by adding the ultra-finely crushed powder of "okara" (50 % grain size: 3.1  $\mu m$ ) to commercial orange juice to a final concentration of 1%. On the other hand, a control product was prepared by adding dry "okara" crushed with the Pinmill type crusher (50 % grain size: 93  $\mu m$ ) to commercial orange juice to a final concentration of 1%. The results obtained in the sensory test by the panel are summarized in Table 14.

(Evaluation: 1(bad) - 5(excellent))

[Table 14]

		Results of Sensory Test				
	Feeling in Throat	Roughness	Feeling in Drinking			
Control Product	1.5	1.2	1.2			
Test Product	4.2	4.5	4.5			

As shown in Table 14, the ultra-finely crushed powder of "okara" (50 % grain size : 3.1  $\mu$ m) is evaluated to be superior to the dry "okara" crushed with the Pinmill type crusher(50 % grain size:93  $\mu$ m) in the sensory test.

(3) Ice cram: A test product was prepared in a conventional manner by mixing fresh cream 200 ml, milk 100 ml, roasted soybean germ crushed ultra-finely (50 % grain size: 3.4 mm) 6 g, sugar

30 g, two pieces of egg yolk, and vanilla essence 0.35 ml. The resulting nice-smelling ice cream had no roughness and gave completely the same feeling on our tongue as commercially available ice cream comprising no roasted soybean germ crushed ultra-finely.

## Example 10

A culture medium (40 ml, pH 7.3) comprising 5.0 g of the ultra-finely crushed powder of soybean hull (50 % grain size: 6.0  $\mu$ m)obtained in Example 7, glucose 0.1 g, KH<sub>2</sub>PO<sub>4</sub> 0.4g, MgSO<sub>4</sub> .7H<sub>2</sub>O 0.05 g, soy milk 130 mg, FeSO<sub>4</sub> .7H<sub>2</sub>O 1 mg, VB<sub>1</sub> 500  $\mu$ g and biotin 500  $\mu$ g was put into a shaking flask (500 ml), capped with a cotton cap, sterilized at 120 °C for 30 min and subjected to a shaking culture (35 °C, 113 rpm) for one month.

No growth of bacteria was observed, showing that the sterilization had been sufficiently done. On the other hand, in the case where the ultra-finely crushed powder of soybean hull (50 % grain size: 95  $\mu m)$  was used instead of the above ultra-finely crushed powder of the present invention in the same way, the growth of bacteria was observed. This result shows that the sterilization was not sufficiently done.

#### Example 11

Various kinds of soybean hull (50 g each) having different 50 % grain size were suspended in water (300 ml), and stirred at a room temperature for 30 min to give water soluble extract. Upper liquid layer was taken out with centrifugation, lyophilized and weighed to give a yield of the extract. The results are shown in Table 15.

[Table 15]

Soybean Hull 50 % Grain Size	Yield of Extract	
6 μm	1 4%	
37 μm	10%	
95 μm	8%	
1~3 mm	5%	

As shown in Table 15, the soybean hull powder crushed ultra-finely up to 50 % grain size of 6.0 µm according to the present invention has been significantly improved in the yield of the extract compared with the powder having a larger grain size.

# Example 12

The ultra-finely crushed powder of soybean hull (50 % grain size: 3.4  $\mu m)$  obtained in Example 7 (26 (parts)), corn protein zein 19 (parts), salt 0.5 (parts), ethanol 33 (parts) and water 22 (parts) were mixed well, shaped in a cast and baked at 150 °C for 5 min to give a tray of 10 cm in diameter, 3 cm high, and 2.5 mm thick. The resulting tray had a smooth surface and enough strength to be used as a tray for food.

For comparison, the soybean hull crushed with the Pinmill type crusher five times (50 % grain size: 12 µm) was used in the same composition and way as above to give a comparative tray. The comparative tray was inferior to the above tray in brightness and strength.

Accordingly, it is shown that the ultra-finely crushed powder (50 % grain size: 3.4  $\mu m$ ) is more advantageous as filler for the production of the food tray than the conventionally used powder (50 % grain size: 12  $\mu m$ ).

The same of the property of the same of th

# Industrial Applicability

As shown by the above examples, the physicochemical properties that are not sufficiently apparent in the whole tissues have appeared in the fractions according to the present invention.

Accordingly, the present fraction as such may be used, for example, as food stuff for various kinds of food (confectionery, bread, desserts such as ice cream, source, retort pouched food, oil and fat food, emulsified food, liquid food, dietary fiber-enriched food, low-calorie food, low-fat food, nutritional component-enriched food, animal food product and marine product and the like), feed, and material for chemical products. The fraction may be also used as a starting material for effectively extracting specific components such as oil and fat or protein based on the difference in the contents of chemical components among the fractions.

The ultra-finely crushed powder according to the present invention has various excellent properties when used as food stuff, such as smooth feeling on tongue, water-holding capacity, and suspendibility in beverage. Furthermore, it shows excellent properties in extracting the contents therein, excellent properties as the component in a culture medium for bacteria in fermentation and brewing, such as excellent sterilization efficiency and transportation efficiency, and excellent properties as the filler for shaping the containers.

#### What is claimed is:

- A method for producing a fraction derived from the fine structure portions of a specific tissue of oilseeds or cereals, comprising crushing and classifying material of the specific tissue.
- A method according to Claim 1 wherein the specific tissue is hull.
- A method according to Claim 2 wherein the hull is selected from the group consisting of soybean hull, corn outer hull, rape seed hull, sesame seed hull and husk.
- A method according to Claim 1 wherein the specific tissue is germ.
- A method according to Claim 4 wherein the germ is selected from the group consisting of soybean germ, corn germ, wheat qerm, and rice germ.
- A method according to Claim 1 wherein the specific tissue is oil and fat extraction cake or protein extraction cake.
- 7. A method according to Claim 6 wherein the oil and fatextraction cake or protein-extraction cake is selected from the group consisting of defatted rice bran, corn germ cake, dry "okara" or soybean pulp, rape seed cake and sesame seed cake.
- A method according to one of Claims 1-7 wherein the classification is carried out based on the difference in specific volume or grain size of the crushed fine structure.
- A method according to one of Claims 1-7 wherein the material is the fraction obtained by the method according to one of Claims 1-7.
- 10. A fraction derived from the fine structure portions of a specific tissue of oilseeds or cereals, which is obtained by the method according to one of Claims 1-9.
- 11. An ultra-finely crushed powder having 50 % grain size of  $6 \mu m$  or less, which is obtained from the material of a specific tissue of oilseeds or cereals.

- 12. An ultra-finely crushed powder according to Claim 11 wherein the material is selected from the group consisting of soybean hull, dry "okara" or soybean pulp, soybean germ, corn germ cake, corn outer hull, whole soybean, defatted soybean, rapeseed cake, and wheat germ.
- 13. A method for producing ultra-finely crushed powder having 50 % grain size of 10 µm or less, comprising crushing material of the specific tissue of oilseeds or cereals in a dry state.
- 14. A method for producing ultra-finely crushed powder according to Claim 14 wherein the crushing is carried out in the dry state by using high-speed compressed air or high-speed compressed gas.
- 15. Food stuff comprising the ultra-finely crushed powder according to Claim 11 or 12, or the ultra-finely crushed powder obtained by the method according to Claim 13 or 14.
- 16. Culture medium for bacteria comprising the ultra-finely crushed powder according to Claim 11 or 12, or the ultra-finely crushed powder obtained by the method according to Claim 13 or 14.
- 17. Filler for shaping containers comprising the ultrafinely crushed powder according to Claim 11 or 12, or the ultra-finely crushed powder obtained by the method according to Claim 13 or 14.

#### ABSTRACT

The purpose of the present invention is to classify tissues of oilseeds and cereals into the fractions of fine structure portions so that physicochemical properties that are not sufficiently apparent in the whole tissues may clearly appear.

The purpose of the present invention is to provide ultra-finely crushed powder that is much more advantageous in the following aspects:

- properties as food stuff such as smoothness, water holding capacity, suspension-state holding capacity;
- 2)sterilization efficiency of the suspension and transportation efficiency in the pipe line when used as material for fermentation;
- extraction efficiency of various components contained in the ultra-finely crushed powder;
- reaction efficiency with enzymes, digestion and absorption efficiency, and feed efficiency when used as feed component;
- 5) quality of containers such as a food tray having a good biodegradablity, which comprises the ultra-finely crushed powder of the present invention as filler.

The present invention relates to a method for producing a fraction derived from the fine structure portions of a specific tissue of oilseeds or cereals, comprising crushing and classifying material of the specific tissue.

The present invention relates to an ultra-finely crushed powder having 50 % grain size of 6 µm or less, which is obtained from the material of a specific tissue of oilseeds or cereals.

The present invention relates to a method for producing ultra-finely crushed powder having 50 % grain size of 10  $\mu$ m or less, comprising crushing material of the specific tissue of oilseeds or cereals in a dry state.

Fig. 1

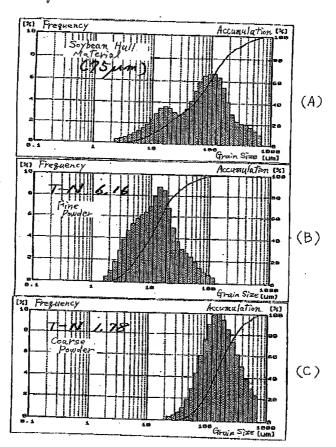
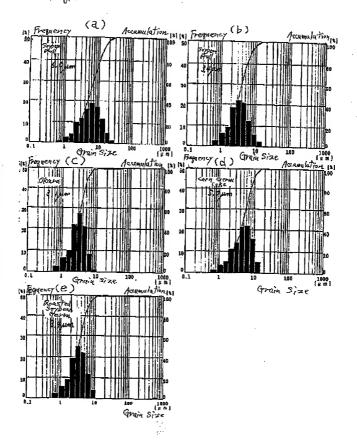


Fig. Z



8/97

# Declaration and Power of Attorney For Patent Application

# 特許出願宣言書及び委任状

# Japanese Language Declaration

# 日本語宣言書

下記の氏名の発明者として、私は以下の通り宣言します。	As a below named inventor, I hereby declare that:
私の住所、私書箱、国籍は下記の私の氏名の後に記載された通りです。	My residence, post office address and citizenship are as stated next to my name.
下記の名称の発明に関して請求範囲に記載され、特許出願している発明内容について、私が最初かつ唯一の発明者(下記の氏名が一つの場合) もしくは最初かつ共同発明者(下記の名称が複数の場合)であると信じています。	I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled.  METHOD FOR CLASSIFYING SPECIFIC TISSUE OF  OILSEEDS OR CEREALS AND FINELY CRUSHED  POWDERS
□ 本書に添付されています。 □ 本書に添付されています。 □ 本書に添付されています。 □ 大切際出原番号または特許協定条 対切際出原番号を とし、 □ (該当する場合) に訂正されました。	the specification of which  is attached hereto.  It was flied on May 4, 2001  as United States Application Number or  PCT International Application Number  09/807,997 and was amended on  (if applicable).
私は、特許請求範囲を含む上記訂正後の明細書を検討し、内容 を理解していることをここに表明します。	I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.
私は、連邦規則法典第37編第1条56項に定義されるとおり、特許 資格の有無について重要な情報を開示する義務があることを認 めます。	I acknowledge the duty to disclose information which is material to patentability as defined in Title 37, Code of Federal Regulations, Section 1.56.
Page :	1 of <u>4</u>

# Japanese Language Declaration (日本語宣言書)

私は、米国法典第35編119条 (a) - (d) 項又は365条 (b) 項に 基づき下記の、米国以外の国の少なくとも一方国を指定してい る特許協力条約365 (a) 項に基づく国際出願、又は外国での特 許出願もしくは発明者証の出願についての外国優先権をここに 主張するとともに、優先権を主張している、本出順の前に出願 された特許または発明者証の外国出願を以下に、枠内をマーク することで、示しています。

Prior Foreign Application(s) 外国での先行出願 10/314150

10/314150	JAPAN
(Number)	(Country)
(番号)	(国名)
10/336688	JAPAN
(Number)	(Country)
(番号)	(国名)

監私は、第35編米国法典119条 (e) 項に基づいて下記の米国特許 監顧規定に記載された権利をここに主張いたします。

(Application No.) (出願番号)

(Filing Date) (出願日)

私は、下記の米国法典第35編120条に基づいて下記の米国特別 出願に記載された権利、又は米国を指定している特許協力条約 655条(c)に基づく権利をここに主張します。また、本出願の各 676条(回)に基づく権利をここに主張します。また、本出願の各 677条(国)を発した。 規定された方法で先行する米国特計出願に開示されていない限 での先行米国出願書提出日以降で本出願書の日本国内また は材許協力条約国際提出日までの期間中に入手された、連邦規 別法典第37編1条56項で定義された特許資格の有無に関する重要 で簡繁について開示義務があることを認識しています。

PCT/JP99/06159 (Application No.) (出願番号) 5 November 1999 (Filing Date) (出顧日)

(Application No.) (出願番号) (Filing Date)

私は、私自信の知識に基づいて本宣言書中で私が行なう表明が 真実であり、かつ私の入手した情報と私の信じるところに基づ く表明が全て真実であると信じていること、さらに故意になさ れた虚偽の表明及びそれと同等の行為は米国法典第18編第1001 条に基づき、罰金または拘禁、もしくはその両方により処罰され ること、そしてそのような故意による虚偽の声明を行なえば、 出願した、又は既に許可された特許の有効性が失われることを 認識し、よってここに上記のごとく宣替を或します。 I hereby claim foreign priority under Title 35, United States Code, Section 119 (a)-(d) or 365(b) of any foreign application(s) for patent or inventor's certificate, or Section 365(a) of any PCT International application which designated at least one country other than the United States, listed below and have also identified below, by checking the box, any foreign application for patent or inventor's certificate, or PCT International application having a filing date before that of the application on which priority is claimed.

	Priority · 優先村	
5 November 1998	X	
(Day/Month/Year Filed)	Yes	No
(出願年月日)	はい	いいえ
27 November 1998	ほい,	□
(Day/Month/Year Filed)	Aes	No
(出願年月日)	►	いいえ

I hereby claim the benefit under Title 35, United States Code, Section 119(e) of any United States provisional application(s) listed below.

(Application No.) (Filing Date) (出願番号) (出願日)

I hereby claim the benefit under Title 35, United States Code, Section 120 of any United States application(s), or Section 365(c) of any PCT International application designating the United States, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT International application in the manner provided by the first paragraph of Title 35, United States Code Section 112, I acknowledge the duty to disclose information which is material to patentability as defined in Title 37, Code of Federal Regulations, Section 156 which became available between the filling date of the prior application and the national or PCT International filling date of application.

(Status: Patented, Pending, Abandoned) (現況:特許許可済、係属中、放棄済)

(Status: Patented, Pending, Abandoned) (現況:特許許可済、係属中、放棄済)

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

## Japanese Language Declaration (日本語宣言書)

委任状:私は下記の発明者として、本出願に関する一切の手続き を米特許商標局に対して遂行する弁理士または代理人として、 下記の者を指名いたします。 (弁護士、または代理人の指名及び登録番号を明記のこと)

(第三以降の共同発明者についても同様に記載し、署名すること)

書類送付先

POWER OF ATTORNEY: As a named inventor, I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and transact all business in the Patent and Trademark Office connected therewith: (list name and registration number)

OBLON, SPIVAK, McCLELLAND, MAIER & NEUSTADT, P.C.

(Supply similar information and signature for third and subsequent

Norman F. Oblon, Reg. No. 24.518; Marvin J. Spivak, Reg. No. 24.913; C. Irvin McClelland, Reg. No. 21.124; Gregory J. Maier, Reg. No. 25.599; Arthur I. Neustadt, Reg. No. 22.824; Richard D. Kelly, Reg. No. 22.757; James D. Hamilton, Reg. No. 28.421; Eckhard H. Kuesters, Reg. No. 29.093; Charles L. Ghotz, Reg. No. 26.935; William E. Beaumin, Reg. No. 30.936; Jean-Paul Lavalleye, Reg. No. 31.451; Stephen G. Baxter, Reg. No. 32.684; Richard L. Treanor, Reg. No. 36.329. Steven P. Weihrouch, Reg. No. 32.828-450n T. Goolkasian, Reg. No. 26.914; Richard L. Treanor, Reg. No. 36.239. J. Derek Mason, Reg. No. 30.011; Carl E. Schlier, Reg. No. 34.426; Steven P. Kulbaski, Reg. No. 34.648; Richard A. Neifeld, Reg. No. 35.239; J. Derek Mason, Reg. No. 35.270; Surinder Sacher, Reg. No. 34.426; Jeffrey B. McIntyre, Reg. No. 36.887; William T. Enos, Reg. No. 35.2128; Michael E. McCabe, Jr., Reg. No. 37.182; Bradley D. Lytle, Reg. No. 40.073; and Michael R. Casey, Reg. No. 40.294, With full powers of substitution and recontinual.

Send Correspondence to:

		1765 JEFFERSON DAVIS HIGHWAY ARLINGTON, VIRGINIA 22202 U.S.A.
直接電話連絡先: (名前及び電話番号)		Direct Telephone Calls to: (name and telephone number) (703) 413-3000
単独発明者または第一の共同発明者の氏名		Full name of sole or first joint inventor Youchi OZAWA
発明者の署名	日付	Inventor's signature Date  Souchi Causa July 4, 200
<b>建</b>	1-4	Residence Kanagawa, Japan
国籍	,	Citizenship Japanese
郵便の宛先		Post Office Address C/o Food Research & Developmentaboratories, Ajinomoto Co., Inc.
-		1-1, Suzuki-cho, Kawasaki-ku, Kawasaki-shi, Kanagawa 210-8681 JAPAN
第二の共同発明者の氏名		Full name of second joint inventor, if any Teruo YOSHIDA
第二の共同発明者の署名	日付	Second joint Inventor's signature Date I nwo Joshi da // Jul 2001
住所	20	Residence Kanagawa, Japan
国籍	No. 2"	Citizenship Japanese
郵便の宛先		Post Office Address c/o Food Research & Development Laboratories, Ajinomoto Co., Inc.
		1-1, Suzuki-cho, Kawasaki-ku, Kawasaki-shi, Kanagawa 210-8681 JAPAN

ioint inventors.)

Page 3 of 4

# Japanese Language Declaration (日本語宣言書)

第三の共同発明者の氏名			Full name of third joint inventor, if any Yasuchi MORINAGA
第三の共同発明者の署名	日付	,/	Third joint Inventor's signature Date Jun 26 200
住所		7/1	Residence Kanagawa, Japan
国籍		()	Citizenship Japanese
郵便の宛先			Post Office Address C/o Food Research & Developm Laboratories, Ajinomoto Co., Inc.
			1-1, Suzuki-cho, Kawasaki-ku, Kawasaki-shi, Kanaqawa 210-8681 JAPAN

		Full name of fourth joint inventor, if any Goro UENO	
日付	1	Fourth joint Inventor's signature Date July	, 20
	(h)		
	X	Citizenship Japanese	
		Post Office Address c/o Ajinomoto Oil Mills Co Inc.	,
		7-14, Daikoku-cho, Tsurumi-ku, Yokohama-si Kanagawa 230-0053 JAPAN	hi.
	日付		Goro UENO  Fourth joint pyentor's signature  Residence Ranaggwa, Japan  Citteenship Japanese  PostOffice Address C/O Ajinomoto Oil Mills Co Inc.  7-14, Dalkoku-cho, Tsurumi-ku, Yokohama-si

第五の共同発明者の氏名			Full name of fifth joint inventor, if any Akihiro NAKATANI
第五の共同発明者の署名	日付	0	Fifth joint Inventor's signature Date July 125
二住所 C		1	Residence Kanagawa, Japan
//国籍		<u></u>	Citizenship Japanese
郵便の宛先			Post Office Address c/o Ajinomoto Oil Mills Co., Inc.
			7-14, Daikoku-cho, Tsurumi-ku, Yokohama-shi, Kanagawa 230-0053 JAPAN

第六の共同発明者の氏名		Full name of sixth joint inventor, if any	
第六の共同発明者の署名	日付	Sixth joint Inventor's signature	Date
住所		Residence	
国籍		Citizenship	
郵便の宛先		Post Office Address	

(第六またはそれ以降の共同発明者に対しても同様な情報および署名を提供すること。)

(Supply similar information and signature for third and subsequent joint inventors.)